

We claim:

1. A process for producing solid preparations of at least one
5 water-soluble, sparingly water-soluble or water-insoluble
active compound suitable for the food and animal feed sectors
or for pharmaceutical and cosmetic applications by
- a) dissolving or dispersing at least one of the
10 abovementioned active compounds in an aqueous molecular
dispersion or colloidal dispersion of a proteinaceous
protecting colloid,
- b) flocculating the proteinaceous protecting colloid
15 together with the active compound out of the dispersion
and
- c) separating off the flocculated solid from the water and
20 any solvents additionally used and subsequently
converting them into a dry powder.
2. A process as claimed in claim 1 for producing solid
25 preparations of at least one sparingly water-soluble or
water-insoluble active compound suitable for the food and
animal feed sectors or for pharmaceutical and cosmetic
applications, wherein, in process step a), at least one of
the abovementioned active compounds is dispersed in an
30 aqueous molecular dispersion or colloidal dispersion of a
proteinaceous protecting colloid.
3. A process as claimed in claim 2, wherein the dispersion step
a) is the production of a suspension of at least one solid
35 active compound in an aqueous molecular dispersion or
colloidal dispersion of a proteinaceous protecting colloid.
4. A process as claimed in claim 3, wherein the suspension
40 produced in process step a) is ground before the
flocculation.
5. A process as claimed in claim 2, wherein the dispersion in
stage a) comprises the following steps:

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- 5 a₁) dissolving one or more sparingly water-soluble or water-insoluble active compounds in a water-miscible organic solvent or a mixture of water and a water-miscible organic solvent or
- 5 a₂) dissolving one or more sparingly water-soluble or water-insoluble active compounds in a water-immiscible organic solvent and
- 10 a₃) mixing the solution obtained by a₁) or a₂) with an aqueous molecular dispersion or colloidal dispersion of a proteinaceous protecting colloid, the hydrophobic phase of the active compound being produced as nanodisperse phase.
- 15 6. A process as claimed in claim 5, wherein, when process step a₂) is being performed, the water-immiscible solvent is distilled off before flocculating the protecting colloid.
- 20 7. *Claim 1*
A process as claimed in ~~one of claims 1 to 6~~, wherein, in process step b), the flocculation is initiated by setting the pH of the dispersion to a value which is in the range of the isoelectric point of the protein used as protecting colloid.
- 25 8. *Claim 1*
A process as claimed in ~~one of claims 1 to 7~~, wherein the protecting colloid is casein or a caseinate.
9. *Claim 1*
A process as claimed in ~~one of claims 1 to 8~~, which involves the production of carotenoid-containing dry powders.
- 30 10. A process as claimed in claim 9 for producing dry powders comprising carotenoids selected from the group consisting of astaxanthin, β -carotene, β -apo-8'-carotenal, β -apo-8'-carotenic acid ethyl ester, canthaxanthin,
- 35 citranaxanthin, echinenone, lutein, lycopene and zeaxanthin.
11. A process as claimed in claim 9, wherein
- 40 a) one or more carotenoids are dissolved in a water-miscible organic solvent, or a mixture of water and a water-miscible organic solvent, at temperatures above 30°C,
- 45 b) the resultant solution is mixed with an aqueous casein solution or caseinate solution,

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- 5 c) the casein or caseinate is flocculated out of the dispersion together with the carotenoid at a pH of the dispersion which is in the region of the isoelectric point of casein or caseinate,
- d) the flocculated solid is separated off from the water and solvent and dried.
- 10 12. A solid preparation of at least one water-soluble, sparingly water-soluble or water-insoluble active compound suitable for the food and animal feed sectors or for pharmaceutical and cosmetic applications and obtainable by a process as defined in ~~one of claims 1 to 11.~~ *claim*
- 15 13. A solid preparation as claimed in claim 12 comprising at least one sparingly water-soluble or water-insoluble active compound suitable for the food and animal feed sectors or for pharmaceutical and cosmetic applications.
- 20 14. A solid preparation as claimed in either of ~~claims 12 or 13~~ having an active compound content of from 0.1 to 80% by weight.
- 25 15. A solid preparation as claimed in ~~either of claims 13 or 14~~ which is a carotenoid-containing dry powder.
- 30 16. A dry powder as claimed in claim 15 comprising carotenoids selected from the group consisting of astaxanthin, β -carotene, β -apo-8'-carotenal, β -apo-8'-carotenic acid ethyl ester, canthaxanthin, citranaxanthin, echinenone, lutein, lycopene and zeaxanthin.
- 35 17. The use of the solid preparation as defined in claim 12 as additive to foods, animal feeds, pharmaceuticals and/or cosmetic preparations.
18. The use as claimed in claim 17, wherein the solid preparation is used in the form of an oil *B* suspension.
- 40 19. The use of the solid preparation as defined in claim 12 for producing oily suspensions.
- 45 20. An oily suspension comprising, as disperse phase, at least one water-soluble, sparingly water-soluble or water-insoluble active compound which is enclosed by one or more protecting colloids and is suitable for the food and animal feed sectors or for pharmaceutical and cosmetic applications, with the

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proviso that the oily suspension comprises no water-soluble vitamins.

21. An oily suspension as claimed in claim 20 comprising, as
5 disperse phase, solid preparations of at least one
water-soluble, sparingly water-soluble or water-insoluble
active compound suitable for the food and animal feed sectors
or for pharmaceutical and cosmetic applications which is
obtainable by
- 10 a) dissolving or dispersing at least one of the
abovementioned active compounds in an aqueous molecular
dispersion or colloidal dispersion of a proteinaceous
protecting colloid,
- 15 b) flocculating the proteinaceous protecting colloid
together with the active compound out of the dispersion
and
- 20 c) separating off the flocculated solid from the water and
any solvents additionally used and subsequently
converting them into a dry powder.
- 25 22. An oily suspension as claimed in ~~either of claims 20 or 21~~
having an active compound content of from 0.1 to 50% by
weight, based on the total amount of oily suspension.
- 30 23. An oily suspension as claimed in ~~one of~~ claims 20 to 22
comprising, as active compound, at least one carotenoid
selected from the group consisting of astaxanthin,
β-carotene, β-apo-8'-carotenal, β-apo-8'-carotenic acid ethyl
ester, canthaxanthin, citranaxanthin, echinenone, lutein,
lycopene and zeaxanthin.
- 35 24. A carotenoid-containing oily suspension as claimed in claim
23 having a water content of from 0.1 to 6% by weight.
- 40 25. A carotenoid-containing oily suspension as claimed in ~~either~~
~~of claims 23 or 24~~, wherein the mean particle size of the
solid phase is in the range from 0.1 to 100 μm.
- 45 26. A carotenoid-containing oily suspension as claimed in ~~one of~~
claims 23 to 25, wherein at least one carotenoid is present
in amorphous or partially amorphous form.

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27. A carotenoid-containing oily suspension as claimed in ~~one of~~ claims 23 ~~to 26~~, which comprises at least one proteinaceous protecting colloid or a modified starch as protecting colloid.
28. An oily suspension comprising, as disperse phase, solid preparations of at least one water-soluble, sparingly water-soluble or water-insoluble active compound suitable for the food and animal feed sectors or for pharmaceutical and cosmetic applications which are obtainable by
- a) dissolving or dispersing at least one of the abovementioned active compounds in an aqueous molecular dispersion or colloidal dispersion of a proteinaceous protecting colloid,
- b) flocculating the proteinaceous protecting colloid together with the active compound out of the dispersion and
- c) separating off the flocculated solid from the water and any solvents additionally used and subsequently converting them into a dry powder.
29. An oily suspension as claimed in claim 28 having an active compound content of from 0.1 to 50% by weight, based on the total amount of oily suspension.
30. An oily suspension as claimed in ~~either of~~ claims 28 ~~or 29~~ comprising as active compound at least one carotenoid selected from the group consisting of astaxanthin, β -carotene, β -apo-8'-carotenal, β -apo-8'-carotenic acid ethyl ester, canthaxanthin, citranaxanthin, echinenone, lutein, lycopene and zeaxanthin.
31. A process for producing a carotenoid-containing oily suspension as defined in claim 23, which comprises
- a) grinding a dry powder comprising at least one carotenoid enclosed by one or more protecting colloids in at least one oil to a mean particle size of from 0.1 to 100 μ m or
- b) grinding a dry powder comprising at least one carotenoid enclosed by one or more protecting colloids without using a continuous phase to a mean particle size of from 0.1 to

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100 μ m and then suspending the ground particles in at least one oil or

- 5 c) grinding a carotenoid-containing suspension comprising, as solid phase, at least one carotenoid enclosed by one or more protecting colloids and, as dispersion medium, water or a mixture of water and a water-miscible solvent to a mean particle size of from 0.1 to 100 μ m, then
10 freeing the solid phase from the water or water/solvent mixture and suspending the resultant ground particles in at least one oil.
- 15 32. A process as claimed in claim 31, wherein the oil is an edible oil liquid at 20°C.
33. A process as claimed in claim 31, wherein the oil is a hard fat solid at 20°C.
- 20 34. The use of the oily suspension as defined in ~~one of claims 20 to 30~~ for producing food supplements and as additive to animal feeds, foods, pharmaceutical and cosmetic preparations.
- 25 35. The use as claimed in claim 34 wherein the oily suspension comprises carotenoid.
36. The use as claimed in ~~either of claims 34 or 35~~ as feed additive in animal nutrition. **B**
- 30 37. The use as claimed in claim 36 for incorporation into feed pellets.
38. The use as claimed in claim 36 for application to food pellets.
- 35 39. The use as claimed in claim 38, wherein the feed pellets are charged with the oily suspension at reduced pressure.
- 40 40. A food supplement, animal feed, food or pharmaceutical or cosmetic preparation comprising an oily suspension as defined in ~~one of claims 20 to 30~~.

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Production of solid preparations of water-soluble, sparingly water-soluble or water-insoluble active compounds

5 Abstract

A process is described for producing solid preparations of at least one water-soluble, sparingly water-soluble or water-insoluble active compound suitable for the food and animal feed sectors or for pharmaceutical and cosmetic applications. In addition, the invention relates to oily suspensions in which these preparations are present as disperse phase, and to the use of these preparations as additive to animal feeds, foods, pharmaceuticals and cosmetic preparations.

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